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12. ABSTRACT (Maximum 200 words)

Initial structural characterization of germanium-silicon epitaxial layers for heterojunction internal photoemission and multiple quantum well infrared detectors has been performed. Cross sectional TEM of multiple quantum wells has been used to confirm period and well thickness measurements performed by other techniques. No defects were observed by TEM in these layers. A variety of other layers including multiple quantum wells on relaxed buffers and heterojunction internal photoemission defector structures have been characterized by defect etching and X- ray diffraction.

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Structural Characterization of Epitaxial Layers for Infrared Detectors

Annual Report AFOSR Contract F49620-92-J-0285

June 1, 1992- May 31, 1993

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AEOSRIR 93 0597

Research Objectives

This contract is an Augmentation Award for Science and Engineering Research Training (AASERT) grant associated with a primary contract concerning the growth of epitaxial heterostructures for infrared detectors. The specific objective of this contract is to perform structural characterization of germanium-silicon heterostructures grown by UHV/CVD.

This contract supports one graduate student in the Materials Science and Engineering (MSE) department at Carnegie Mellon (S. Vyas) and also provided support for a summer undergraduate student (T. Knight). The activities of these two students are summarized below.

Status of the Research Effort

Course Requirement and Ph.D. Qualifier - S. Vyas

The MSE program at Carnegie Mellon has an intensive first year which emphasizes coursework, culminating in the Ph.D. qualifying exam in June. As a result, research activities were necessarily limited although some initial studies were conducted as described in the following section.

We are pleased to report that S. Vyas has completed the required coursework with excellent grades (6 As and 1 B). He received a very strong pass in the Ph.D qualifying exam in early June. Having completed these academic requirements, he has begun intensive research with objectives discussed below.

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 In view of the course requirements, our objective in the first year has been to provide an introduction to the techniques to be used in subsequent research. In the course of this, structural characterization necessary for the project was performed and some samples for subsequent investigation were grown and characterized by X- ray diffraction. This work is detailed in the following sections.

TEM Characterization of undoped multiple quantum well samples

A variety of undoped multiple quantum well samples used in a previous study ["Characterization of Undoped Multiple Quantum Well Structures," R. Misra, R. Strong, D.W. Greve, and T.E. Schlesinger, (to be published in *J. Vac. Sci. Technol.*).] were examined by cross-sectional TEM. The objectives were to (1) verify other measurements of well thickness and periodicity; (2) look for defects and the possible onset of islanding; and (3) to provide an introduction to the use of the TEM.

Under the direction of a postdoctoral student (Christina DeSouza), samples for cross sectional TEM were prepared by sawing followed by wet etching and finally ion milling. The samples were examined in a JEM 120 under dark field imaging conditions. No signs of islanding or other defects were apparent from the photographs, and all interfaces were planar. From the photographs, the period and quantum well thickness were measured. The results are summarized in Table I and compared with X-ray diffraction measurements of the same samples.

| | X- ray diffraction | | X-TEM | |
|-------------|--------------------|----------------|--------|----------------|
| Ge fraction | period | well thickness | period | well thickness |
| 0.19 | 206 Å | 57 Å | 200 Å | 46 Å |
| 0.19 | 198 Å | 50 Å | 190 Å | 51 Å |
| 0.19 | 185 Å | 45 Å | 201 Å | 35 Å |
| 0.19 | 170 Å | 27 Å | 168 Å | 36 Å |

Table I. Comparison of results of X-ray diffraction and cross sectional TEM measurements on undoped multiple quantum well structures (20 wells, (100) silicon substrates).

The results are in generally good agreement with X- ray diffraction measurements although the agreement is better with the period than with the quantum well thickness. To some degree this is due to the difficulty of determining the edges of the wells. We expect to refine the technique and perform additional comparisons with X- ray diffraction in future samples.

The absence of apparent islanding is reasonable considering the relatively low germanium fraction (x = 0.19) and thin layers. We are concerned about the onset of islanding in thicker layers of higher germanium fraction used in heterojunction internal photoemission detectors. This will be a subject of future investigation.

The absence of visible defects is encouraging (and of course expected in light of the good photoluminescence). We will be moving to more sensitive techniques for the quantification of dislocations (misfit and threading) such as wet defect etching and X- ray topography. We are also planning to examine graded buffer layers with large defect concentrations in order to provide a baseline.

Multiple Quantum Wells on Graded Buffers

Graded buffer layers are necessary in order to grow strain-symmetrized superlattices. In addition, there has been recent interest in understanding the relaxation process in these layers as particularly low densities of threading dislocations have been reported. The graded buffer layers reported here were grown together with S. Vyas who wrote the growth program and supervised the growth. Details of the growths are given in Table II.

| sample | number of graded layers | graded layer thickness | MQW periods | QW thickness | barrier thickness |
|--------|-------------------------|---------------------------|-------------|--------------|-------------------|
| GB1,2 | 9 | 400 Å | 20 | 50 Å | 50 Å |
| GB3,4 | 21 | 100 Å | 20 | 50 Å | 50 Å |
| GB5,6 | 21 | 100 Å | 40 | 30 Å | 30 Å |

Table II. Details of growth of multiple quantum well structures on relaxes buffers (intended thicknesses). The barrier concentration was 10% Ge and the well concentration 30% Ge.

The samples were characterized by S. Vyas by ordinary θ - 2θ X- ray diffraction. Typical data is shown in Fig. 1. The results show that the expected periodicity of the multiple quantum well layers is in fact observed. The observation of sharp lines and multiple satellite peaks suggests that strain relaxation has taken place in the buffer layer and that the multiple quantum wells are uniformly strained. We plan to perform double- crystal X- ray diffraction measurements on these samples in the near future. Characterization by photoluminescence and atomic force microscopy (in collaboration with Digital Instruments, Co.) is in progress. TEM examination is also planned.

Relaxation and Islanding in Thicker Layers

Growth with a non-planar surface morphology (or islanding) was observed in early investigations by Bean and coworkers and has recently received intensive investigation by laser light scattering and atomic force microscopy [A.J. Pidduck et al., *Thin Solid Films* 222, 78 (1992)]. We are concerned about the possibility that this may occur in layers necessary for heterojunction internal photoemission detectors. These detectors require relatively high germanium fraction, high boron concentration, and relatively thick layers ($\approx 300 \text{ Å}$, much thicker than multiple quantum well detectors). Thus islanding should be most pronounced in these layers, and may be influenced to an unknown degree by high boron concentrations.

S. Vyas is presently investigating the surface morphology of layers appropriate for HIP detectors using a variety of techniques. We have grown a variety of structures with varying germanium fraction (0.10- 0.32) and boron concentration (undoped to 8×10^{19} cm⁻³). An investigation of the effect of growth temperature is also planned for the future.

These layers have already been characterized by wet defect etching and preparation of samples for cross-sectional TEM is presently in progress. We also plan characterization by X-ray topography and other analytic techniques (SIMS and RBS). The goal is to correlate the results of materials analysis with the characteristics of detector structures made from these layers. This correlation will lead to the establishment of optimal growth conditions for the HIP detectors.

Calculations and Measurements on Multiple Quantum Wells (T. Knight)

T. Knight was employed as a summer research student from approximately June 1 to August 15, 1992. He subsequently was admitted to Carnegie Mellon for graduate study and is now working on an integrated readout structure for infrared detector arrays.

During the summer of 1992, he worked on two main projects. Experimental work involved setting up apparatus for far- infrared detector measurements and interfacing the insturments to a computer. This has been completed and the apparatus is now in use.

Theoretical work involved the calculation of the band diagrams of doped quantum well structures for infrared detectors. This work led to considerable insight into the desired layer parameters.

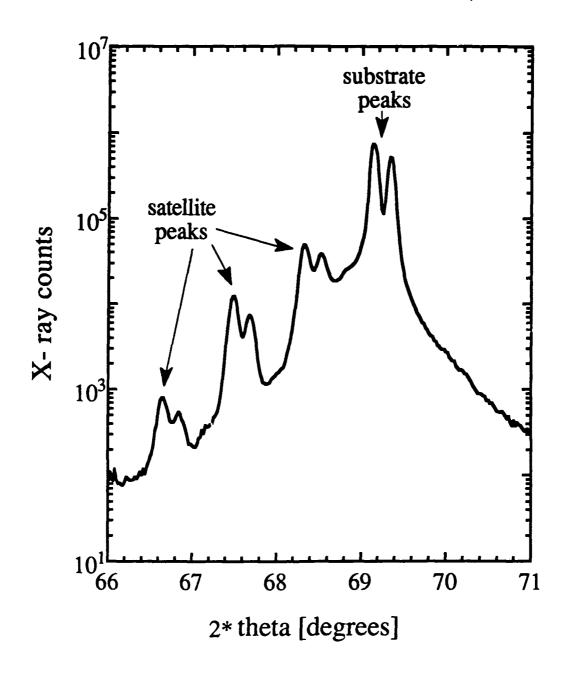


Figure 1. X- ray diffraction scan of multiple quantum well structure on relaxed buffer layer (sample GB1)

Publications

(Note: this section lists all publications supported by the parent grant .F49620-92-J-0155)

Review Article

"Growth of Epitaxial Germanium-Silicon Heterostructures by Chemical Vapor Deposition," D.W. Greve, *Materials Science and Technology* **B18**, 22 (1993).

Journal Articles

"Incorporation of Boron into UHV/CVD- Grown Germanium-Silicon Epitaxial Layers," D.W. Greve and M. Racanelli, J. Electron. Mater. 21, 593 (1992).

"Uniformity of Ge_xSi_{1-x} Epitaxial Layers Grown by Ultra-High Vacuum Chemical-Vapor Deposition," D.W. Greve, G. McLaughlin, M.A. Capano, and M. Racanelli, (to be published in *Appl. Phys. Lett.*).

"Photoluminescence and X-Ray Diffraction Study of Highly Uniform Si and Ge_xSi_{1-x} Epitaxial Layers" D.W. Greve, R. Misra, T.E. Schlesinger, and G. McLaughlin, *Thin Solid Films* 222, 46 (1992).

"Photoluminescence Characterization of UHV/ CVD Grown Multiquantum Wells," R. Misra, D.W. Greve, and T.E. Schlesinger, (to be published in *J. Electron. Mater.*).

"Characterization of Undoped Multiple Quantum Well Structures," R. Misra, R. Strong, D.W. Greve, and T.E. Schlesinger, (to be published in *J. Vac. Sci. Technol.*).

Conference Proceedings

"Kinetics of Thermal Cleaning for Silicon and Germanium-Silicon Epitaxy," M. Racanelli and D.W. Greve, Proc. Second. Int'l. Symposium on Cleaning Technology in Semiconductor Device Manufacturing, pp. 461-468, (The Electrochemical Society, Pennington, NJ, 1992).

"Comparison of Mesa-Etched and Ion-Implanted Ge_xSi_{1-x} Heterojunction Bipolar Transistors," D.W. Greve and M. Racanelli, (to appear in *Proc. 1992 Spring MRS, Symposium on Defect Engineering in Semiconductor Growth, Processing, and Device Technology*).

"Growth and Characterization of Ge_xSi_{1-x} Multiple Quantum Well Structures," D.W. Greve, R. Misra, M.A. Capano, and T.E. Schlesinger, *Proc. 1992 Spring MRS, Symposium on Mechanisms of Epitaxial Growth*, pp. 365- 370, (Materials Research Society, Pittsburgh, PA, 1992).

"Electrical and Structural Properties of Cobalt Annealed on Silicon-Germanium Epilayers," G. Sarcona, F. Lin, M.K. Hatalis, A.F. Cserhati, E. Austin, and D.W. Greve, (to appear in *Proc.* 1992 Fall MRS).

Personnel

Faculty

D.W. Greve, Professor ECE, principal investigator S. Mahajan, Professor MSE, co- advisor of S. Vyas

Graduate Students

Sanjay Vyas

(M.S. student MSE, supported by AASERT grant, US citizen)

Summer Undergraduate Student

Thomas Knight

(Senior undergraduate at Penn State, supported by AASERT grant, US citizen)

Interactions

Wright Laboratory, WPAFB

M.A. Capano- high resolution X- ray diffraction W. Mitchel- spectrally resolved photoconductivity

Air Force Institute of Technology

R.L. Hengehold- cathodoluminescence and UV- excited photoluminescence

Lehigh University

M.K. Hatalis- investigation of cobalt silicides

Jet Propulsion Laboratory

E. Fossum-heterojunction internal photoemission FET (integrated detector/readout; supported by Caltech President's fund)

Papers at meetings (upcoming)

"UHV/ CVD Growth of Germanium- Silicon," D.W. Greve, R. Misra, R. Strong, and T.E. Schlesinger, (invited paper, American Vacuum Society Fall Meeting, Orlando, FL, November, 1993).

Papers at meetings (February 1, 1992-January 31, 1993)

"Comparison of Mesa-Etched and Ion-Implanted Ge_xSi_{1-x} Heterojunction Bipolar Transistors," D.W. Greve and M. Racanelli, 1992 Spring MRS, Symposium on Defect Engineering in Semiconductor Growth, Processing, and Device Technology, San Fransciso, CA.

"Thickness and Compositional Uniformity of Epitaxial Layers Grown by UHV/ CVD, M.A. Capano, D.W. Greve, and M. Racanelli, 1992 Spring MRS, Symposium on Mechanisms of Epitaxial Growth, San Fransciso, CA.

"Growth and Characterization of Ge_xSi_{1-x} Multiple Quantum Well Structures," D.W. Greve, R. Misra, M.A. Capano, and T.E. Schlesinger, 1992 Spring MRS, Symposium on Mechanisms of Epitaxial Growth, San Fransciso, CA.

"Characterization of Doped Ge_xSi_{1-x} Multiple Quantum Well Structures for Far- IR Detectors," R. Misra, D.W. Greve, R. Strong, and T.E. Schlesinger, (Symposium on Infrared Detectors-Materials, Processing, and Devices, Spring 1993 MRS Meeting).*

"Kinetics of Epitaxial Layer Growth from Silane on (100 Silicon, D.W. Greve, (Symposium on Common Themes and Mechanisms of Epitaxial Growth, Spring 1993 MRS Meeting).*

"Polycrystalline Germanium- Silicon- Growth and Applications," D.W. Greve (invited paper, Integrated Prossing for Micro- and Opto- Electronics, European MRS, May, 1993).

"Photoluminescence Characterization of UHV/ CVD Grown Multiquantum Well Structures," R. Misra, D.W. Greve, and T.E. Schlesinger, 1992 Electronic Materials Conference, Boston, MA.

"Characterization of Undoped Multiple Quantum Well Structures, "R. Misra, R. Strong, D.W. Greve, and T.E. Schlesinger, 1992 Fall Meeting of the American Vacuum Society, Chicago, IL.

"Electrical and Structural Properties of Cobalt Annealed on Silicon-Germanium Epilayers," G. Sarcona, F. Lin, M.K. Hatalis, A.F. Cserhati, E. Austin, and D.W. Greve, 1992 Fall MRS, Boston, MA.

"Photoluminescence and X- ray Diffraction Study of Highly Uniform Silicon and Ge_xSi_{1-x} Epitaxial Layers, invited paper, 1992 Spring E- MRS, Strasbourg, France.

Seminars

"Growth and Characterization of Ge_xSi_{1-x} Multiple Quantum Well Structures," Wright Laboratory, Wright- Patterson AFB, October 30, 1992.

"Growth and Characterization of Ge_xSi_{1-x} Multiple Quantum Well Structures," Department of Electrical Engineering, Notre Dame University, November 11, 1992.

"Applications of UHV/ CVD- Grown Silicon and Germanium- Silicon, Electronic Imaging Laboratory Seminar, Xerox Palo Alto Research Center, November 13, 1992.

Other

Session Co- Chair, Silicon - Based Heterostructures II- Quantum Devices, Fall 1992 AVS Meeting, Chicago, IL.

Inventions/ Patent Disclosures

None.